B. Manufacturing/Analytical Techniques/Quality Assurance of Mentholated Cigarettes

1. Use and Control of Menthol in Cigarette Products

This section will discuss the actual menthol application process, the operational procedures, the sampling and testing procedures, and the analytical methodology for testing and reporting menthol content in tobacco cigarettes, packaging, and smoke.

2. The Application of Menthol

The process used for producing mentholated cigarettes is common to most of the tobacco industry and involves adding a menthol solution to an alcohol-based top-dressing solution. (Currently, RJRT uses a propylene glycol-based top-dressing solution.(1) Mentholated foil is also being investigated for domestic use. RJRTI has adopted mentholated foil on Salem KS in Malaysia.) The top-dressing solutions are sprayed onto cut filler in a top-dressing drum. The application level of menthol is higher than the level specified for the packaged product to allow for system losses during application, storage, and transport of the tobacco prior to manufacturing the cigarettes. The majority of system losses (on the order of 4% to 6%) are probably stack losses (losses of alcohol/propylene glycol and menthol to the atmosphere via exhaust air flow from the process).

Once the tobacco is top-dressed, the cut filler is transfered to bulk blenders or placed in tubs until it is used in the manufacturing of cigarettes (approximately 12-24 hours after top-dressing). The use of bulk blenders has decreased the variation in pack menthol levels, however, the use of tubs is still dominant.

When the tub system is used, lots of 5 tubs are dumped into a redresser drum for mixing prior to pneumatic feeding to the making machines. In some instances, the pneumatic transfer system is not used and tubs are taken directly to the cigarette maker for hand feeding or "black-hose" feeding to the hopper on the cigarette maker. A benefit of the tub-storage system is the ability to "mix-off" tubs of high and low % moisture (or menthol) in the redresser when high variation among tubs is detected by Production personnel. Therefore, this system provides a potential method for correcting a processing error if the error has been detected. A weakness of this system is the potential for mixing up the tubs fed to the redresser with the result that the wrong blend is sent to the making floor. This is not a rare occurrence. It is caused by inadequate controls in the handling and shuffling of large numbers of tubs - about 10,000 in Whitaker Park alone. Currently, this number is being reduced. However, when many small brands must be run in one factory the problem is compounded. Not only is there greater likelihood for mix-up but the small volume of tobacco required makes the processing run short. This creates significant variations in moisture levels and top-dressing levels at the beginning and end of each run. Until planned bulking systems are in full use, these processing problems will be encountered. More diligent monitoring, labeling, and handling procedures will reduce the errors due to product mix-up.

The variations regularly observed in pack-menthol level, from $\pm$ 5% to $\pm$ 10% of target level, are probably entirely due to the application system used. W. W. Barnhardt and R. H. Cundiff did a comprehensive study on SALEM KS in 1966. They examined the variation of % tobacco menthol at various points in the process and found a C.V. (coefficient of variation is the percentage of variation about the mean) of $\pm$ 16% on the conveyor belt after the top-dressing drum. The C.V. decreased to $\pm$ 10% for tobacco in tubs before storage and $\pm$ 6-8% after 48 hours of tub storage. The C.V. of menthol-in-the-pack, for all packs sampled hourly for all shifts for one

week, was in the range of ± 6-7%. Currently, RJR is striving to reduce the C.V. for all mentholated brands to a level within ± 5%.

In their study, Barnhardt and Cundiff also determined the influence of tobacco temperature at the top-dressing drum on % tobacco menthol level. A comparison of the processes at two factories indicated that when tobaccos were top-dressed at a high temperature (~ 121°F) versus a lower temperature (~ 92°F) a significant loss in % menthol level occurred (~7-10%). Standardization between factories and reductions in the temperature of the cut-filler to be top-dressed will decrease variations in % menthol levels for RJR products.

Mr. R. H. Cundiff has submitted a detailed study of in-plant procedures for top-dressing application which has been prepared by Mr. S. A. Garner. This comprehensive document correctly describes the present manufacturing procedures for top-dressing application and control, as of February 1982. It is part of a booklet describing procedures and methods for preparation and analysis of mentholated products.

3. Analytical Methodology

o Tobacco and Cigarette Components

For many years, the steam distillation procedure has been used to determine the menthol content of tobacco and cigarette components. In this procedure, the distillate was diluted with ethanol and reacted with a reagent to yield a red color. The colorimetric absorbance was then determined with the Robot Chemist. Since December of 1981, menthol determinations have been performed with both the colorimetric and the G-C methods, but recently the G-C method was adopted as the normal method of analysis. (Analytical Method Number 2002-1). A review of the literature which lists published works on methods for separating and determining menthol in tobacco products has been completed. The bibliography is part of the booklet on Procedures and Methods for Mentholated Products at RJR.

o Menthol In Cigarette Smoke

To determine smoke menthol, three unconditioned cigarettes are smoked under standard FTC conditions (35cc puff volume, 2 second duration, 1 puff per minute (frequency)), at room conditions of 60% R.H. and 75°F. Cigarette smoke is adsorbed on filter material (Cambridge pads or similar filters) which is subsequently extracted with chloroform containing an internal standard (anethole). Aliquots of the extract are analyzed for menthol by a G-C method similar to Analytical Method Number 2002-1. The analysis is normally duplicated and the average menthol delivery is reported. The Analytical Method Number for determination of smoke menthol is 1005. Descriptions of the Analytical Methods can be requested from the Analytical Services Division.

o Menthol in Packaging Materials

The analytical procedure for determining menthol in packaging materials requires the extraction of menthol from the packaging material from one pack of cigarettes with methylene chloride. Separation and quantitation of the menthol is performed by G-C. Anethole is used as an internal standard. The development of the procedure and results from selected experiments are described in RDM, 1982, No. 19 by M.S. Uhrig, R.A. Heckman, and N.M. Davis.

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4. Quality Assurance

The only role that Quality Assurance has in menthol application and control is providing routine sampling of cigarettes from production. The procedure is outlined in the booklet on Procedures and Methods for Mentholated Products at RJR.

The sampling procedure is as follows:

(1) One cigarette pack is collected every 1 1/2 hours, 5 times daily from each shift, for a total of 5 packs per shift for each menthol brand. Each pack is collected from a different machine.

(2) As each pack is collected, it is immediately labled with the time sampled, machine number, shift number, department number, 4 digit brand code for Export products not using domestic blend, and date.

(3) Samples from each shift are kept separate and are delivered personnally to QA.

(4) When sample sizes are less than expected, it is due to production schedule.

When problems occasionally develop and analytical results exceed the target value by ± 20% of target, QA will notify the appropriate people in Manufacturing and R&D. Notification will occur whenever excessive deviation is noted - sometimes on a shift-by-shift basis. In some cases, the action limits are held closer than ± 20% for notification.

5. Bibliography

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11. IOM, Menthol Cigarette Protocol, D.R. Pugh, 4/16/82.

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